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(71) Applicant *(for all designated States except US)*: BEDE SCIENTIFIC INSTRUMENTS LIMITED [GB/GB]; Bowburn South Industrial Estate, Bowburn, Durham DH6 5AD (GB).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: ARNDT, Ulrich, Wolfgang [GB/GB]; 28 Barrow Road, Cambridge CB2 2AS (GB). LONG, James, Victor, Percival [GB/GB]; University of Cambridge, Bullard Laboratory, Dept. of Earth Sciences, Madingley Rise, Cambridge CB3 0EZ (GB). DUNCUMB, Peter [GB/GB]; 5A Woollards Lane, Great Shelford, Cambridge CB2 5LZ (GB).

(74) Agent: MURGITROYD & COMPANY; 373 Scotland Street, Glasgow G5 8QA (GB).

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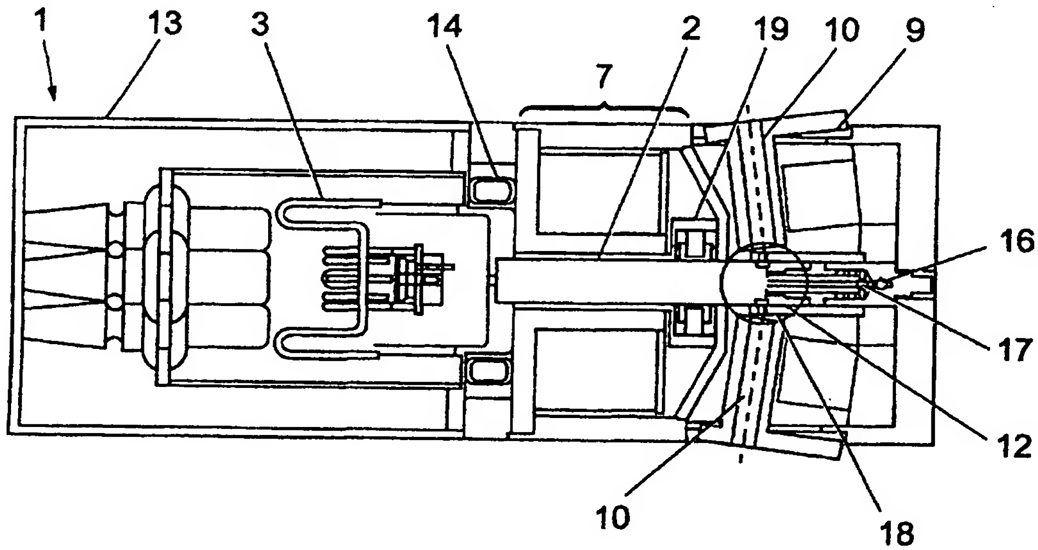
(54) Title: X-RAY GENERATOR



(57) Abstract

An X-ray generator comprises an evacuated and sealed X-ray tube, an electron gun, an X-ray target, an internal electron mask, and an X-ray window consisting of a thin tube of material with low X-ray absorption and high mechanical strength, for example beryllium. The window connects the tube to the target assembly containing the X-ray target. The generator preferably also includes a system for focusing and steering the electron beam onto the target, a cooling system to cool the target material, kinematic mounts to allow precise and repeatable mounting of X-ray devices for focusing the X-ray beam, and X-ray focusing devices of varying configurations and methods. The X-ray generator of the invention produces an X-ray source having a focal spot or line of very small dimensions and is capable of producing a high intensity X-ray beam at a relatively small point of application using a low operating power.

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X-Ray Generator

This invention relates to an X-ray generator and in particular to an X-ray generator suitable to be closely coupled to a focusing X-ray device.

X-ray generators comprise an electron gun, an X-ray target and an X-ray exit window, generally in a sealed vacuum. Prior art generators produce X-ray beams having a relatively large focal spot or line. Many applications require a precisely collimated X-ray beam. To achieve this relatively small apertures are coupled with the generator to restrict beam diameter and divergence, but this results in a large loss of X-ray intensity.

For many applications the most effective way of using the X-rays emitted from the target of an X-ray tube is to form an image of the source, i.e. of the electron focus on the target, on the specimen. For crystallographic applications, it is normally essential that the convergence or divergence of the rays incident on the sample be very small. To maximise the X-ray intensity at the sample the angle of collection at the source should be as large as possible. The combination

of these two requirements implies that the imaging optics should magnify. The sample size determines the maximum useful image size (see Fig. 3). Fig. 3 shows that the ratio of the collecting angle α at the source S to the beam convergence angle β at the image I is equal to the magnification of the focusing collimator or focusing mirror F. In single-crystal diffractometry, for example, the specimen crystal is frequently about 300 μm in diameter. The X-ray source should, therefore, be much smaller than 300 μm.

Maximum power loading of the target, without damage to its surface is greatest when the source is a line focus at a small take-off angle to give a foreshortening of about 10 times.

It is an object of the present invention to provide an X-ray generator which produces an X-ray source having a focal spot or line of very small dimensions. It is a further object of the present invention to provide an X-ray generator capable of producing a high intensity X-ray beam at a relatively small point of application using a low operating power.

According to a first aspect of the invention there is provided an X-ray generator comprising an electron gun, electron focusing means and a target, the electron focusing means being arranged such that the X-ray source on said target may be varied in size and/or shape and/or position.

Preferably the X-ray source on said target may be varied from a small diameter spot to a line of small width.

Preferably the generator further comprises an X-ray

exit window comprising a tube of material with low X-ray absorption and of a small diameter to allow close coupling of X-ray focusing devices.

Preferably the electron focusing means comprises an electron beam focusing means mounted around the X-ray tube. The electron beam focusing means may comprise an x-y deflection system for centring the electron beam in the X-ray tube. The electron beam focusing means may further comprise at least one electron lens, preferably an axially symmetric or round lens, and at least one quadrupole or multipole lens for focusing the electron beam to a line focus. The line focus preferably has an aspect ratio in the range 1:1 to 1:20.

The electron beam lenses may be magnetic or electrostatic and are preferably electronically controlled.

Preferably the material of the exit window has a high mechanical strength and is preferably beryllium. The exit window may form part of the mechanical structure of the X-ray tube and preferably connects the X-ray tube and the target.

Preferably the target is metal, most preferably a metal selected from the group Cu, Ag, Mo, Rh, Al, Ti, Cr, Co, Fe, W, Au. In a preferred embodiment the target is copper. The target surface may be orientated such that the plane of the target surface is perpendicular or at an angle to the axis of the X-ray tube.

The target may comprise a thin metal layer deposited on a thicker substrate of a material with high thermal conductivity. Preferably the substrate material is diamond.

Preferably the generator further comprises a target cooling means. According to a first embodiment the cooling means may comprise means for directing a jet of fluid onto the target, on the opposite side of the target to the side on which the electron beam impinges. The fluid is preferably air or water. According to a second embodiment the cooling means may comprise means for effecting heat transfer by conduction or convection from the target.

Preferably the generator further comprises a deflection means which spatially scans the position of the electron beam over the face of the target.

Preferably the generator further comprises an electron mask having an aperture adapted to align the focal spot of the electron beam.

According to a second aspect of the invention there is provided an X-ray generator comprising an electron gun, an X-ray tube, a target and an X-ray exit window comprising a tube of material with low X-ray absorption and of small diameter to allow close coupling of X-ray focusing devices.

According to a third aspect of the invention the generator according to the first or second aspects is coupled with an X-ray focusing means. The X-ray focusing means preferably comprises a mirror.

The X-ray source according to the invention is designed specifically to be closely coupled to focusing X-ray devices. It is able to produce a focal spot or line of very small dimensions, and thus maximise the benefit of the focusing methods.

The distance from the electron focus to the exit window exterior is very small, and can be as low as 7 mm or less for a reflection target, or less than 1 mm for a foil transmission target.

The X-ray generator according to the invention is compact and provides a sealed tube.

The X-ray generator according to the invention needs only low power because of the efficiency of the collection and subsequent delivery of X-rays to the sample.

The generator achieves a high brilliance, defined as X-ray power per unit area per steradian.

An embodiment of the invention will now be described, by way of example only, with reference to the accompanying figures, where:

Fig. 1 shows a longitudinal section through an X-ray generator according to the invention;

Fig. 2 shows a detail to an enlarged scale of part of the X-ray generator shown in Fig. 1;

Fig. 3 shows the relationship between the size of an X-ray source and the image at a sample; and

Fig. 4 shows the variation in X-ray intensity as an electron beam is scanned across an aperture in front of a target.

With reference to Figs. 1 and 2, the X-ray generator 1 comprises an evacuated and sealed X-ray tube 2, containing the following elements:

- Electron gun 3
- X-ray target 4
- Internal electron mask 5
- X-ray window 6 consisting of a thin tube of material with low X-ray absorption and high mechanical strength, for example beryllium. This window also connects the tube 2 to the target assembly 12 containing the target 4.

The tube 2 is contained within a housing 13. The generator 1 also includes a system 7 for focusing and steering the electron beam onto the target, a cooling system 8 to cool the target material, kinematic mounts 9 to allow precise and repeatable mounting of X-ray devices for focusing the X-ray beam, and X-ray focusing devices 10 of varying configurations and methods. X-ray mirrors 10 are supplied in pre-aligned units so that re-alignment is not necessary after exchange.

The X-ray tube 2 produces a well focused beam of electrons impinging on a target material 4. The electron beam may be focused into a spot or a line, and the dimensions of the spot and line as well as its position may be changed electronically. A spot focus having a diameter falling in the range 1 to 100 μm, generally 5 μm or larger, may be achieved. Alternatively a line focus may be achieved whose width falls in a similar range, having a length to width ratio of up to 20:1.

An electron beam mask of 5 of metal (eg tungsten) in the form of an internal electron beam aperture 11, with suitable dimensions, for example a rectangular slot for the line focus, may be used with suitable feedback and control mechanisms to automatically align the focal spot and to maintain its position on the target, for

example by scanning the electron beam over the aperture 11 and measuring the emerging X-ray intensity.

The electron beam is produced by an electron gun 3, consisting of a Wehnelt electrode and cathode. The cathode may be either:

- a filament of tungsten or alloy, for example tungsten-rhenium, having either a hairpin or a staple shape; or
- an indirectly heated activated dispenser cathode, which may be flat or of other geometry, for example a rod with a domed end.

The dispenser cathode has the advantage of extended lifetime and increased mechanical strength. With a flat surface the dispenser cathode has the further advantage of requiring only an approximate degree of alignment in the Wehnelt electrode.

Primary focus is achieved by an anode at a suitable distance from the electron gun.

A thin tube of material with low X-ray absorption but high mechanical strength and stability, such as beryllium, is used to form the exit window 6 for the emerging X-rays. The tube must exhibit good vacuum seal characteristics. This tube also forms the mechanical connection between the X-ray tube 2 and the target assembly 12. Such an arrangement saves space and complexity in the formation of X-ray windows.

The electron beam from the gun is centred in the X-ray tube 2 by a centring coil 14 or set of quadrupole lenses. Alternatively it may be centred by multipole lenses. The electron beam is focused to a spot of varying diameter. Focusing down to a diameter of less than 5 μm or better may be achieved by an axial lens 7

consisting of either quadrupole, multipole or solenoid type.

The spot focus may be changed to a line focus with a further set of quadrupole or multipole lenses. Lines with an aspect ratio of greater than 10:1 are possible. A line focus spreads the load on the target. When viewed at a suitable angle, the line appears as a spot.

Lenses are preferably magnetic, but may be electrostatic. All the lenses are electronically controlled, enabling automatic and continuous alignment and scanning of the focal spot. Change from spot to line is also automatic, as is the change of beam diameter.

The target 4 is a metal, for example Cu, but it can be another material depending on the wavelength of the characteristic radiation required, for example Ag, Mo, Al, Ti, Rh, Cr, Co, Fe, W or Au. The target 4 is either perpendicular to the impinging electron beam, or may be inclined to decrease the absorption of the emitted X-rays.

The target is cooled either by:

- a jet of cooling fluid (water, air or another fluid) directed onto the rear surface of the target area by cooling nozzle 15; or
- conducted or convected heat transfer from the rear of the target 4.

The cooling fluid is circulated through an inlet 16 and outlet 17.

An increase in cooling efficiency (and hence an increase in the permissible target loading) may be

achieved by the use of a thin metal film of target material deposited on a thicker substrate made from a material with a high thermal conductivity (eg diamond). The target could comprise a thin solid of a single material or it could be laminated with a different material of high thermal conductivity. These targets may be used with different cooling geometries, for example those employing high or low water pressure or forced or natural convection.

Both foil transmission and reflection targets may be used as a target 4.

Integrated mechanical shutters 18 are positioned between the window 6 and the X-ray focusing elements 10, to block the emerging X-ray beam.

The placement of the shutter 18 before the focusing elements 10 protects the surface of the mirror from extended radiation damage.

A compact X-ray detector may be included to monitor and continuously optimise the position of the electron focal spot. This may be a small solid state detector or other X-ray detecting device.

The system encompasses an X-ray focusing device 10 located close to the source to provide a magnified image of the focal spot at controlled varying distances from the source. Options for the X-ray focusing systems are:

1 Micromirrors: use specular reflectivity from a gold or similar coating of highly controlled smoothness (around 10 Å rms), from a circularly symmetric profile.
   - Ellipsoidal profile: gives focused beam of X-

rays (currently 300 μm diameter 600 mm from focal spot). Measured insertion gain of > 150 (could be 250+). Reason for close coupling is so that a large solid angle of radiation may be collected, but also focusing element forms a magnified image of the focal spot at the sample (low beam divergence but high insertion gain)

- Paraboloidal profile: gives a nearly parallel beam (expected gains around 200+)

2 Kirkpatrik-Baez type:

- Bent plates arranged in combinations of elliptical or parabolic or combination
- Allows simple change of mirror profiles to suit different applications

3 Other possibilities:

- Zone plates
- Bragg Fresnel optics
- Multilayer optics

The distance x between the focusing mirror 10 and the source on the target 4 is small, usually lerss than 20 mm, preferably about 11 mm, to ensure close coupling.

<u>Example</u>

A number of copper-target X-ray tubes with focusing collimators were constructed to the same basic specifications shown in the table below.

<u>Table of Specifications</u>

X-ray tube target	Copper, cooled by water or forced air

Source size	15 μm x 150 μm viewed at 6°
Present tube current	0.2 mA at 30 kV
X-ray focusing	Ellipsoidal mirror, gold surface
Source-to-mirror distance	11 mm
Solid angle of collection	8.0 x 10^{-4} sterad
Beam convergence at sample	10^{-3} rad

The cathode is at negative high voltage and the electron gun consists of a filament just inside the aperture of a Wehnelt grid which is biased negatively with respect to the filament. The electrons are accelerated towards the anode which is at ground potential and pass through a hole in the latter and then through a long pipe (tube 2) towards the copper target 4. An electron cross-over is formed between the Wehnelt and anode apertures and this is imaged on the target by the iron-cored axial solenoid 7 which surrounds the vacuum pipe. The best electron focus is obtained when the beam passes very accurately along the axis of the solenoid. Two sets of beam deflection coils 14, which may be iron-cored, are employed in two planes separated by 30 mm, mounted between the anode of the electron gun 3 and the axial solenoid 7 to centre the beam. Between the solenoid 7 and the target 4 is an air-cored quadrupole magnet which acts as a stigmator 19 in that it turns the circular cross-section of the beam into an elongated one. This

quadrupole 19 can be rotated about the tube axis so as to adjust the orientation of the line focus. The beam can be moved about on the target surface 4 by controlling the currents in the four coils of the quadrupole 19.

For a tube power below 2 watts the foil target is adequately cooled by radiation alone, but at higher powers forced-air or water-cooling is necessary. The tube may be operated continuously at 6 watts but the maximum power compatible with low damage to the target surface 4 is still to be established.

Computer simulations show that the loading limit of a water-cooled copper target and a focus of 15 μm x 300 μm is about 20 watts. Experiments suggest that this figure can be somewhat improved upon by increasing the turbulence in the flow of the coolant. Another approach is to sandwich a layer of a material with a very high thermal conductivity between a very thin copper target layer and a cooled copper block. The sandwiched layer may be a Type II diamond layer, and may be sandwiched between a 5 μm thick copper target layer and a water-cooled copper block. Diamond has a thermal conductivity which is up to four times that of copper and our calculations show that its use should allow the permissible power dissipation to be approximately doubled.

The electron source of a micro-focus X-ray tube must have a high brightness to produce gun currents of the order of 1 mA.

An indirectly heated cathode a Few hundred micrometers in diameter may be used. The beam cross-section remains circular until the beam reaches the stigmator

quadrupole while it can be drawn out into a line between 10 μm and 30 μm in width and with a length-to-width ratio up to 20:1. Such an electron source consumes a much lower filament power than the hair-pin tungsten filaments customary for low-power applications; since it operates at a lower temperature, it can have a life of several thousand hours.

The tube is run in a saturated condition in which the current is virtually independent of the filament temperature but is determined by the bias voltage between filament and Wehnelt electrode. This bias voltage is the potential drop produced by the tube current flowing through a high resistor; this form of autobias produces a very stable tube current which is readily controlled by varying the bias resistance.

The electron-optical performance of the tubes has been investigated by fitting some of them with 20 μm thick transmission targets. This allowed pinhole photographs of the focus to be made. A quick way of assessing the focus was to view the magnified shadow cast by a 200- or 400-mesh grid. The electron beam could also be scanned across a rectangular aperture immediately in front to the target. The results are shown in Fig. 4, which shows how the X-ray intensity varies as the electron beam is scanned across the aperture in front of the target. It can be seen that the intensity reaches a peak of about 4000 cps over a range of distance between 60 and 220 micrometres.

The insertion gain of ellipsoidal mirrors was measured. This gain was defined as the ratio of CuKα X-ray flux into the 0.3 mm diameter image of the X-ray source formed at a distance of 600 mm from the source to the flux into the same area without the mirror. Under

these conditions the cross-fire at the sample position is about 1 milliradian. For the best mirrors the insertion gain was 110.

The X-ray intensity obtained as above was also compared with that obtained at the focus of a standard double Franks mirror arrangement used with an Elliot GX-21 rotating anode X-ray generator operated at 2kW. (This is a conventional combination of X-ray tube and collimator for protein crystallography). When the tube according to the invention was operated at below 1 watt, the intensity was only 25 times less than that from the rotating-anode operated at a power 2000 times greater. Further improvements are possible, both in X-ray tube power and in mirror performance. It should be noted that the insertion gain calculated simply on the basis of solid angles of the cone of radiation collected from the source and on the highest values of X-ray reflectivity which have been measured is approximately five times greater than that achieved so far.

These and other modifications and improvements can be incorporated without departing from the scope of the invention.

CLAIMS

1. X-ray generator comprising an electron gun, an X-ray tube, electron focusing means and a target adapted to have an X-ray source formed thereon, the electron focusing means being arranged such that the X-ray source on the target may be varied in size and/or shape and/or position.

2. X-ray generator according to Claim 1, wherein the X-ray source on said target may be varied from a small diameter spot to a line of small width.

3. X-ray generator according to Claim 1 or 2, further comprising an X-ray exit window comprising a tube of material with low X-ray absorption and of a small diameter to allow close coupling of X-ray focusing devices.

4. X-ray generator according to Claim 3, wherein the material of the exit window has a high mechanical strength and is preferably beryllium.

5. X-ray generator according to Claim 3 or 4, wherein the exit window connects the X-ray tube and the target.

6. X-ray generator according to any preceding Claim, wherein the electron focusing means comprises an x-y deflection system for centring the electron beam in the X-ray tube.

7. X-ray generator according to Claim 6, wherein the electron beam focusing means further comprises at least one electron lens, preferably an axially symmetric or round lens, and at least one

quadrupole or multipole lens for focusing the electron beam to a line focus.

8. X-ray generator according to any preceding Claim, wherein the target is a metal foil transmission target, the metal being selected from the group Cu, Ag, Mo, Rh, Al, Ti, Cr, Co, Fe, W, and Au.

9. X-ray generator according to any preceding Claim, wherein the surface of the target impinged upon by the electron beam is orientated such that the plane of the target surface is perpendicular or at an angle to the axis of the X-ray tube.

10. X-ray generator according to any preceding Claim, wherein the target comprises a thin metal layer deposited on a thicker substrate of a material with high thermal conductivity, preferably diamond.

11. X-ray generator according to any preceding Claim, wherein the generator further comprises a target cooling means.

12. X-ray generator according to any preceding Claim, further comprising an electron mask having an aperture adapted to align the focal spot of the electron beam.

13. X-ray generator comprising an electron gun, an X-ray tube, a target and an X-ray exit window comprising a tube of material with low X-ray absorption and of small diameter to allow close coupling of X-ray focusing devices.

14. X-ray generator according to any preceding Claim,

further comprising an X-ray focusing means coupled closely to said target.

15. X-ray generator according to Claim 14, wherein the X-ray focusing means comprises an X-ray mirror whose longitudinal alignment axis is arranged at an angle to the axis of the X-ray tube.

16. X-ray generator according to Claim 15, wherein the angle is between 80° and 90°, preferably about 84°.

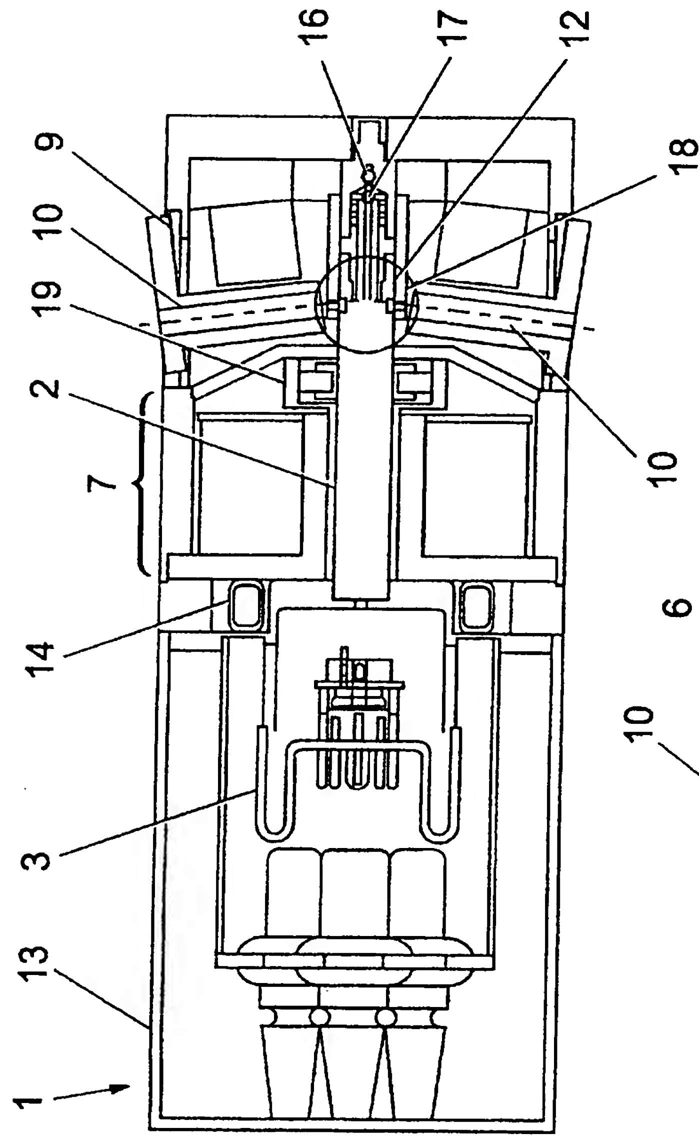

Fig. 1

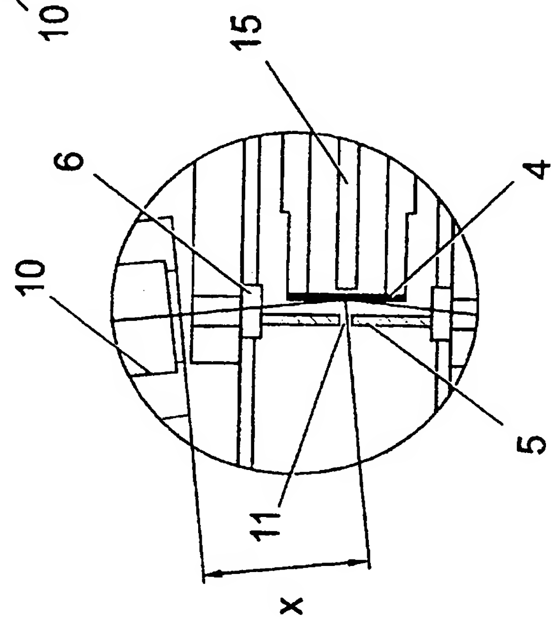

Fig. 2

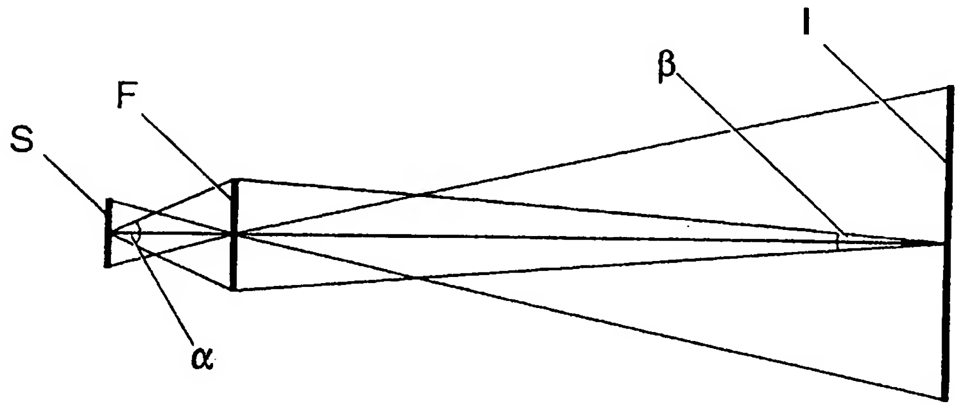
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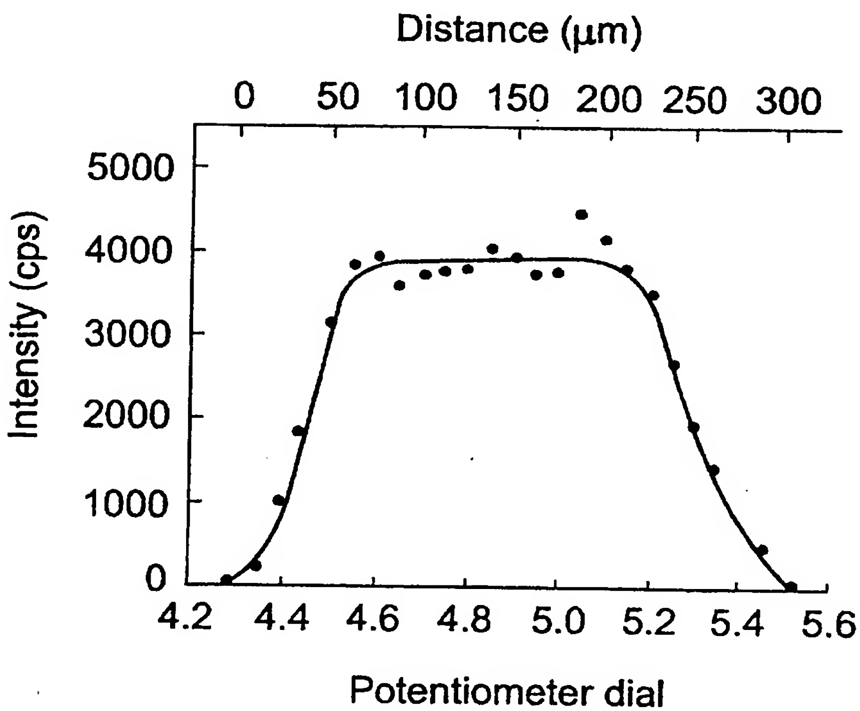
Fig. 3


Distance (μm)
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50
100
150
200
250
300
5000
4000
3000
2000
1000
0
Intensity (cps)
4.2
4.4
4.6
4.8
5.0
5.2
5.4
5.6
Potentiometer dial

Fig. 4

INTERNATIONAL SEARCH REPORT

In. ıational Application No
PCT/GB 97/02580

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 H01J35/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 H01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 732 426 A (SHIMIZU T) 8 May 1973 see figures see column 1, line 50 - column 2, line 43	1-5,9,13
Y		6-8,10, 11,14-16

Y	US 4 827 494 A (KOENIGSBERG WILLIAM D) 2 May 1989 see figure 1 see column 3, line 39 - line 55 ---	6,7
Y	EP 0 319 912 A (WANG CHIA GEE DR) 14 June 1989 see column 5, line 25 - line 38 see column 6, line 19 - line 33 --- -/--	8,10

[X] Further documents are listed in the continuation of box C.

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1

Date of the actual completion of the international search	Date of mailing of the international search report
5 January 1998	13/01/1998

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Colvin, G

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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